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ORIGINAL ARTICLE

Adsorption kinetic and isotherm studies of Azure A on various activated carbons derived from agricultural wastes

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Abstract The present study narrates the eminent role of agricultural wastes as adsorbents *viz.*, Indian almond shell carbon (IASC), ground nut shell carbon (GSC), areca nut shell carbon (ASC), tamarind shell carbon (TSC) and cashew nut shell carbon (CSC) for the removal of Azure A (AA) dye from waste water. Different experimental parameters such as effect of initial concentration, contact time, dose, pH and particle size have been studied. The experimental results were analysed using Freundlich, Langmuir, Temkin, Redlich–Peterson and Dubinin–Radushkevich isotherm models. Different kinetic equations (first order, pseudo first order and pseudo second order) were applied to study the adsorption kinetics of AA on various activated carbons. Surface morphology of the adsorbents before and after adsorption is studied by Scanning Electron Microscopy (SEM). FT-IR studies revealed the presence of functional groups of dye on the adsorbents. It is inferred from the experimental result that the activated carbons (IASC, GSC, ASC, TSC and CSC) from agricultural wastes can be applied as an adsorbent substitute to commercial activated carbon (CAC) in the removal of AA dye from waste water.

1. Introduction

The usage of dyes in textile, paper, paint and cosmetics industries goes on increasing year by year. Consequently the ratio of pollutants increases. The waste water discharged from these industries containing toxic dyes and chemicals pollutes the surface water heavily. (Singh and Srivastava, 2001). The removal of dyes from waste water is an urgent need to protect the environment and aquatic life. Even though different methods

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were employed, adsorption is more efficient than other methods in the removal of dyes from waste water. Commercial activated carbon (CAC) has been widely used as an adsorbent for this purpose. Due to high cost of CAC, low cost adsorbents have to be developed. Many researchers have shown great interest on this part and found the use of low cost activated carbons prepared from agricultural wastes in the removal of dyes. Some of the adsorbents prepared from agricultural wastes were sugarcane bagasse (Azhar et al., 2005; Saad et al., 2010), desert plant (Bestani et al., 2008), hazelnut shells (Ferrero, 2007), oil palm wood (Ahmad et al., 2007), orange peel (Sivaraj et al., 2001), peanut hull (Gong et al., 2005), jackfruit peel (Jayarajan et al., 2011; Hameed, 2009), wheat straw, corncobs, barley husk (Robinson et al., 2002), rice husk (Han et al., 2008; Sharma et al., 2010) and coconut shell (Kannan and Meenakshi sundaram, 2001). Biosorbents like chitosan (Iqbal et al., 2011) and eggshell (Ahmad et al., 2012) were employed to remove dye and metal ions.

The aim and scope of the present study is to ascertain adsorption capacity and removal efficiency of AA dye from waste water on four different adsorbents namely IASC, GSC, ASC, TSC and CSC by the batch adsorption method.

2. Materials and methods

2.1. Chemicals, reagents, adsorbent preparation and activation

Azure A (AA) dye supplied by Loba Chemie, India is used as an Adsorbate and λ_{max} value is 625–634 nm. All the other chemicals used in this study were of analytical grade and used as received.

The adsorbents required for this study are prepared from Indian almond shell, ground nut shell, areca nut shell, tamarind shell and cashew nut shell. They were collected locally and carbonized at 400 °C. Activation was done by treatment with acid, washed and heated at 120 °C for one hour in an air oven. Finally they were sieved to discrete particle sizes. All the solutions and reagents were prepared by double distilled water.

2.2. Instrumentation study

The SEM images of activated carbons were taken from scanning electron microscope (Hitachi S-3400 N). The FT-IR spectra of activated carbons were recorded by a Shimadzu Spectrometer. Hanna pen pH metre was used to study the effect of pH. The activated carbons were separated into different particle sizes with the help of a mechanical siever (Jayant brand, India). The optical density (OD) of dye solution was measured by using an Elico UV–visible spectrophotometer (model: SL207).

2.3. Batch mode adsorption studies

Exactly 50 ml of dye AA solutions of different initial concentrations was prepared in a standard measuring flask from the stock solution. A standard graph is drawn by connecting OD against concentration. The concentration of dye solution before and after adsorption is calculated from the standard graph. The batch mode experiments were conducted at room temperature (30 ± 1 °C). The dye AA solutions of different initial concentrations (C_i = 5–100 ppm) were prepared. Exactly 50 ml of AA solutions of various initial concentrations was taken and treated with dose of adsorbents (cac and all activated carbons = 2.0 g L^{-1}) of fixed particle size (90 micron) at initial pH (7.1) and shaken in a thermostatic mechanical shaker for 30 min with 200 rpm at 30 ± 1 °C. The effect of contact time was studied by keeping initial concentration, dose, pH and particle size constant except time (5–60 min). The effect of dose variation was studied by kept initial concentration, contact time, pH and particle size constant except dose (70–130 mg for all activated carbons and cac). The effect of pH was studied by keeping initial concentration, contact time, dose and particle size constant except pH (2–11 for all activated carbons and cac). The effect of particle size was studied by kept initial concentration, contact time, dose and pH constant except particle size (90–250 micron for all activated carbons). The percentage of AA removal is calculated using the following relationship

$$\text{Percentage removal} = \frac{(C_i - C_f)}{C_i} \times 100 \quad (1)$$

The amount adsorbed in (mg g^{-1}) is calculated using the following relationship.

$$\text{Amount adsorbed } (q_e) = \frac{(C_i - C_f)}{m} \quad (2)$$

C_i and C_f be the initial and final concentrations (mg L^{-1}) of dye respectively and 'm' be the mass of activated carbon (mg L^{-1}).

3. Result and discussion

3.1. Effect of initial concentration

The batch type adsorption studies on the extent of removal of dye AA on activated carbons (IASC, GSC, ASC, TSC and CSC) and CAC were carried out at different initial concentrations (AA on activated carbons = 5–100 ppm and AA on CAC = 350–600 ppm) with a fixed dose (2 g L^{-1}) and contact time 30 min at room temperature (30 ± 1 °C). The percentage removals of dye AA on all the carbons tend to decrease with an

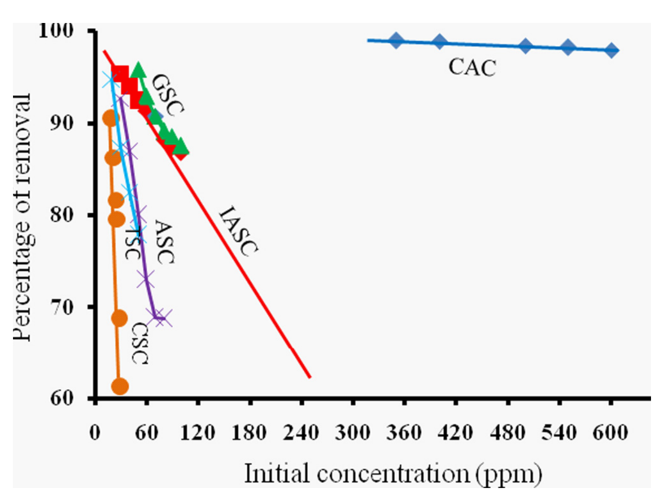

Figure 1 Effect of initial concentration of AA on various activated carbons.

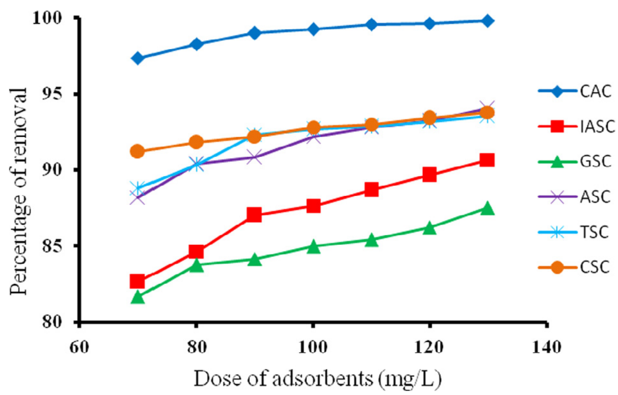

Figure 2 Effect of dose variation of AA on various activated carbons.

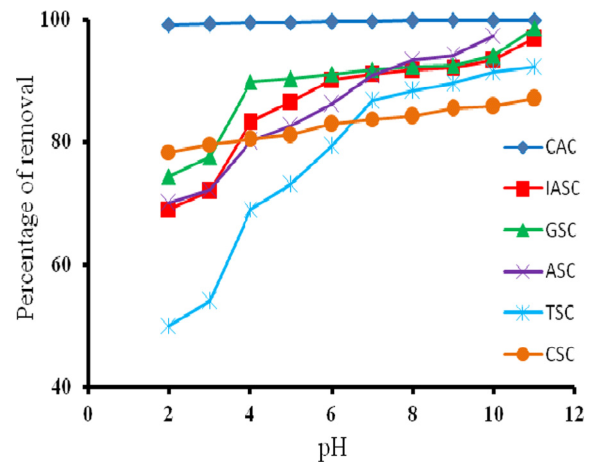

Figure 3 Effect of pH variation of AA on various activated carbons.

increase in the initial concentration (Fig. 1). This may be due to lack of available active sites required for the high initial concentration of AA. At lower concentrations a substantial amount of AA got adsorbed leaving behind more number of active sites on the adsorbents and at higher concentrations lesser number of active sites remained available. Similar results have been reported in the literature on the extent of removal of dyes (McKay, 1983) and metal ions (Kannan 1991).

3.2. Effect of dose of adsorbents

The effect of dose of adsorbents on the extent of removal of dye AA on activated carbons (IASC, GSC, ASC, TSC and CSC) and CAC was studied. The percent removal of AA by activated carbons and CAC increases with an increase in dose of adsorbents (Fig. 2). This may be due to an increase in the availability of surface active sites.

3.3. Effect of pH

The effect of pH on adsorption process for AA on activated Carbons (IASC, GSC, ASC, TSC and CSC) and CAC was studied at different pH values (2–11). At low pH, the surfaces of activated carbons may become positively charged due to excess of H^+ ions in solution. The electrostatic repulsion between AA and surfaces of adsorbents decreases the amount of AA adsorbed. At high pH, the surfaces of activated carbons may become negatively charged due to excess of OH^- ions in solution. Consequently electrostatic attraction between cationic dye and anionic surfaces of activated carbons increases the amount of AA adsorbed (Fig. 3). The maximum percentage removal of AA occurs at pH 10–11.

3.4. Effect of particle size

The effect of particle size of activated carbons (IASC, GSC, ASC, TSC and CSC) to remove AA by adsorption was studied by varying the particle size as 90, 125, 150, 212 and 250 micron. The amount of AA adsorbed increases with a decrease in particle size of the activated carbons (Fig. 4). When the particle size decreases, the surface area increases.

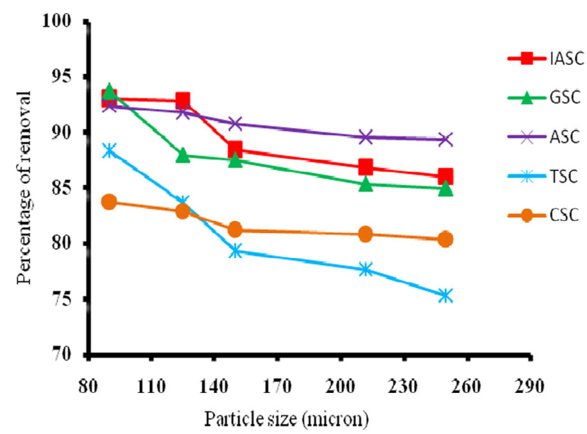

Figure 4 Effect of particle size variation of AA on various activated carbons.

3.5. Adsorption isotherm

The data obtained from the study of AA on Activated carbons (IASC, GSC, ASC, TSC and CSC) and CAC by batch adsorption were applied with Freundlich, Langmuir, Temkin, Redlich–Peterson and Dubinin–Radushkevich isotherms (Table 1).

3.5.1. Freundlich isotherm

$$\text{Freundlich isotherm}\ \left(\log\ \frac{x}{m}\right) = \log\ K + \left(\frac{1}{n}\right)\log\ C_e \qquad (3)$$

In the above equation $\log K$ value is the intercept and $(1/n)$ value is the slope (Freundlich,1906). The plot of $\log C_e$ against $\log x/m$ gives straight line (Fig. 5) and the correlation coefficient (r value) indicates the applicability of this isotherm. These values are shown in Table 1. The value of $1/n$ lower than unity shows that AA is adsorbed by the activated carbons (IASC, GSC, ASC, TSC and CSC) and CAC.

Table 1 Adsorption isotherm for the removal of AA by activated carbons and CAC.

Isotherm	Parameters	Adsorbents					
		CAC	IASC	GSC	ASC	TSC	CSC
Freundlich	Slope ($1/n$)	0.730	0.278	0.437	0.262	0.281	0.276
	Intercept ($\log k$)	0.371	0.022	0.631	0.741	0.676	0.853
	r-value	0.999	0.901	0.981	0.964	0.971	0.967
Langmuir	Slope ($1/Q_0$)	0.001	0.008	0.010	0.016	0.021	0.045
	Intercept ($1/Q_0b$)	0.006	0.045	0.033	0.063	0.054	0.021
	r-value	0.997	0.986	0.984	0.982	0.988	0.998
	Q_0 (mg g^{-1})	834	114.2	106.8	59.05	46.29	21.88
	b (g L^{-1})	0.183	0.196	0.277	0.266	0.393	0.999
	$R_L = 1/(1 + C_0b)$	0.013	0.092	0.056	0.069	0.079	0.040
Temkin	A (L g^{-1})	1.505	1.868	3.600	5.850	5.715	1.710
	B	197.9	25.45	21.59	10.30	8.988	2.190
	r-value	0.996	0.986	0.957	0.948	0.970	0.859
Redlich–Peterson	K_R	153.9	22.47	39.75	15.74	18.24	46.38
	αR	0.536	0.562	0.813	0.758	0.921	1.407
	β	0.891	0.872	0.722	0.759	0.639	0.978
	r-value	0.999	0.990	0.996	0.969	0.953	0.998
Dubinin–Radushkevich	X'_m(mg g^{-1})	582.0	62.15	75.88	46.06	31.05	21.13
	E(kJmol^{-1})	5.60	10.38	9.69	9.044	15.54	14.98
	K' (mol^2 kJ^{-2})10^{-7}	−1.6	−4.6	−5.3	−6.1	−2.1	−2.2
	r-value	0.967	0.924	0.842	0.849	0.838	0.928

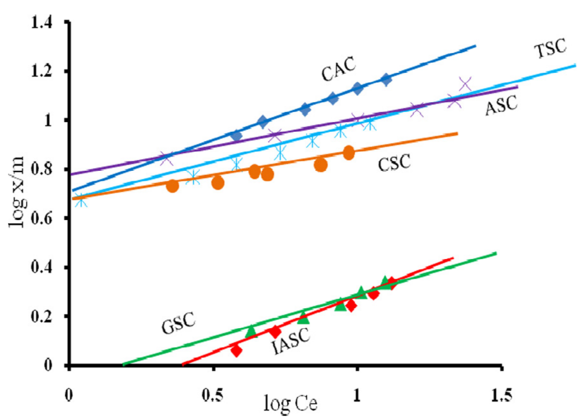

Figure 5 Freundlich isotherm plot for adsorption of AA on various activated carbons.

3.5.2. Langmuir isotherm

$$\text{Langmuir isotherm}\left(\frac{C_e}{q_e}\right) = \left(\frac{1}{Q_0 b}\right) + \left(\frac{C_e}{Q_0}\right) \tag{4}$$

q_e – Amount of dye adsorbed per unit mass of adsorbent (mg g^{-1}).
x – Weight of dye adsorbed.
m – Weight of adsorbent.
Q_0 – Monolayer adsorption capacity (mg g^{-1}).
C_e – Equilibrium concentration (ppm).
b – Langmuir constant.

The Langmuir isotherm plot for AA on activated carbons (IASC, GSC, ASC, TSC and CSC) and CAC is found to be linear (Fig. 6) with correlation coefficient (r) closer to unity indicating that the adsorption data are best fitted to this isotherm (Langmuir 1918). The feasibility of the process is expressed in terms of separation factor R_L which is given by the equation.

$$R_L = \frac{1}{(1 + bC_i)} \tag{5}$$

b – Langmuir constant.
C_i – Initial concentration of dye.

The separation factor R_L indicates the nature of isotherm and the feasibility of adsorption process as favourable. The R_L values are found to be in the range of 0–1, indicating that the adsorption process is favourable (Senthilkumar et al., 2005). R_L values for AA on activated carbons are 0.040–0.092 and AA on CAC is 0.013 which indicates that the adsorption process is favourable. The order of adsorption capacities (Q_0) of all carbons is as follows. CAC > IASC > GSC > ASC > TSC > CSC.

3.5.3. Temkin isotherm

The linearised form of Temkin isotherm is given below (Allen et al., 2004).

$$q_e = B \ln A + B \ln C_e \tag{6}$$

where, $B = RT/b$, b is the Temkin constant related to heat of sorption (J/mol), A is the equilibrium binding constant (L g^{-1}), R the gas constant (8.314 J mol^{-1} K^{-1}) and T is the absolute temperature (K). The constants A and B are derived from the graph drawn by connecting q_e against $\ln C_e$. The values of the parameters are given in Table 1. The correlation coefficient r values for activated carbons (IASC, GSC, ASC, TSC and CSC) and CAC indicate the possibility of this isotherm.

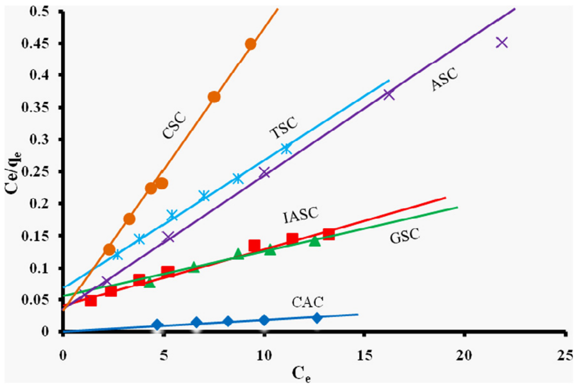

Figure 6 Langmuir isotherm plot for adsorption of AA on various activated carbons.

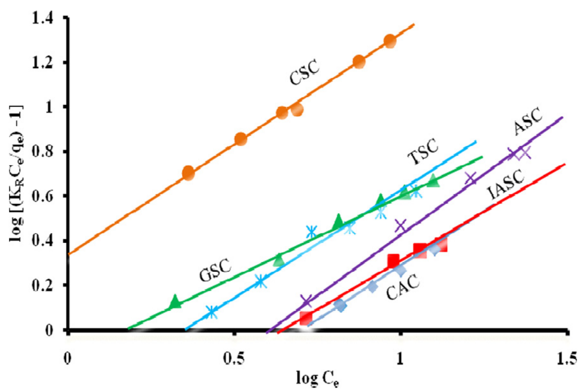

Figure 7 Redlich–Peterson isotherm plot for adsorption of AA on various activated carbons.

3.5.4. Redlich–Peterson isotherm

The linearised form of Redlich–Peterson equation is given below

$$\log\left[\left(\frac{K_R C_e}{q_e}\right) - 1\right] = \beta \log C_e + \log(\alpha R) \quad (7)$$

$$K = Q_0 b (\mathrm{L\ \ g^{-1}})$$

Q_0 – Langmuir monolayer adsorption capacity (mg g^{-1}); b – Langmuir constant (L mg^{-1}); (αR) – R–P isotherm constant; β – Exponent of R–P isotherm; C_e – equilibrium/liquid phase concentration of solute (mg L^{-1}), m – mass of adsorbent (g L^{-1}); x – amount of solute adsorbed by solid adsorbent (mg L^{-1}); q_e – equilibrium amount of solute adsorbed (mg g^{-1}). By plotting the values of $\log[(K_R C_e/q_e) - 1]$ against $\log C_e$, the values of (αR) and β can be determined from the intercept [log (αR)] and the slope (β) respectively (Fig. 7). The adsorption data perfectly fitted to this isotherm.

3.5.5. Dubinin-Radushkevich isotherm

$$q = X'_m \exp(-K' \in^2) \quad (8)$$

where, $\in = RT \ln(1 + 1/C_e)$, q is the amount adsorbed per unit mass of adsorbent (mg g^{-1}), X'_m is the adsorption capacity (mg g^{-1}), C_e is the equilibrium concentration of adsorbate in solution (mg L^{-1}), R is the universal gas constant, K' is the constant related to the adsorption energy (mol^2 kJ^{-2}) and T is the temperature (K). The linearised form of D–R equation is given below

$$\ln q = \ln X'_m - K' \in^2 \quad (9)$$

The plot of ln q against $\in^2$ gives a straight line. The values of X'_m and K' are calculated from the intercept and slope. From the K' value, the mean energy of adsorption E (kJ mol^{-1}) is calculated.

$$E = (-2K')^{-0.5} \quad (10)$$

The mechanism of adsorption is estimated by the magnitude of E. In this study the value of E is <16 (kJ mol^{-1}) for activated carbons (IASC, GSC, ASC, TSC and CSC) and CAC which indicates that adsorption may be dominated by particle diffusion.

3.6. Kinetics of adsorption

The following equations were used to study the kinetics of adsorption of AA on all carbons and the results are given in Table 2.

Table 2 Statistical results of kinetic equations for the removal of AA by activated carbons and CAC.

Kinetic equation	Parameters	Adsorbents					
		CAC	IASC	GSC	ASC	TSC	CSC
First order	k (min^{-1})	0.037	0.217	0.193	0.561	0.497	0.245
	r-value	0.854	0.819	0.950	0.778	0.904	0.887
Natarajan and Khalaf	k (min^{-1})	0.293	0.144	0.073	0.110	0.121	0.117
	r-value	0.924	0.999	0.950	0.961	0.939	0.939
Venkobachar and Bhattacharya	k (min^{-1})	0.390	0.225	0.273	0.130	0.209	0.267
	r-value	0.965	0.960	0.980	0.959	0.982	0.968
Pseudo first order	k (min^{-1})	0.036	0.029	0.052	0.034	0.175	0.267
	r-value	0.972	0.995	0.981	0.869	0.980	0.968
Pseudo second order	k (min^{-1})	24.52	3.394	2.794	2.492	1.588	1.266
	r-value	0.999	0.999	0.999	0.999	0.999	0.999
Intra particle diffusion	k_p	0.079	0.019	0.017	0.045	0.022	0.009
	r-value	0.979	0.994	0.968	0.941	0.968	0.973
	Intercept	19.23	2.180	2.670	1.840	1.200	0.986

Table 3 Comparison of calculated and experimental q_e values for adsorption of AA by activated carbons and CAC.

Kinetic equation	Parameters	Adsorbents					
		CAC	IASC	GSC	ASC	TSC	CSC
Pseudo first order	q_e exp. (mg g^{-1})	398.4	56.1	46.8	43.2	27.1	21
	q_e cal. (mg g^{-1})	3.32	2.547	2.708	10.37	4.398	1.534
	R^2 value	0.969	0.9695	0.991	0.734	0.935	0.954
	Δq (%)	35.32	33.09	34.95	37.59	35.02	37.21
Pseudo second order	q_e exp. (mg g^{-1})	398.4	56.1	46.8	43.2	27.1	21
	q_e cal. (mg g^{-1})	398.8	56.30	46.95	44.16	27.47	21.15
	R^2 value	0.999	0.999	0.999	0.999	0.999	0.999
	Δq (%)	0.293	0.696	0.688	1.315	0.898	1.455

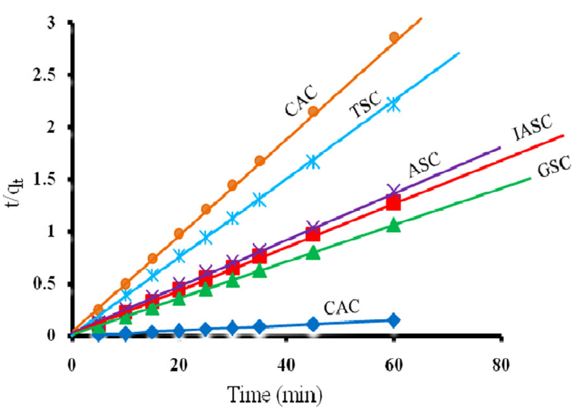

Figure 8 Pseudo second order plot for adsorption of AA on various activated carbons.

First order equation

$$\left(\frac{1}{q_t}\right) = \left(\frac{K}{q_{max}}\right)\left(\frac{1}{t}\right) + \left(\frac{1}{q_{max}}\right) \tag{11}$$

Natarajan and Khalaf equation

$$\log\left(\frac{C_i}{C_t}\right) = \left(\frac{k}{2.303}\right)t \tag{12}$$

Bhattacharya and Venkobachar equation

$$\log\ [1 - U(t)] = -\left(\frac{K_{ad}}{2.303}\right)t \tag{13}$$

where, C_i and C_t are respectively, the concentration of dye (in ppm) at time zero and at time t; q_e and q_t are the amount of dye adsorbed per unit mass of the adsorbent (in mg g^{-1}), respectively at equilibrium time and at time t, and q_{max} is the maximum adsorption capacity (in mg g^{-1}), k and k_{ad} are the adsorption rate constants (min^1) and U(t) = $[(C_i - C_t)]/(C_i - C_e)]$; C_e = concentration of dye (ppm) at equilibrium (optimum) contact time. The experimental data are applied in the above equations and found to be statistically significant. The adsorption data are tested in pseudo first order equation to study the adsorption of solid/liquid systems. Pseudo first order equation

$$\log\ (q_e - q_t) = \log\ q_e - \left[\frac{K_{ad}}{2.303}\right]t \tag{14}$$

k_{ad} (min^{-1}) is the rate constant of the adsorption and q_t and q_e are the amount adsorbed at time t and at equilibrium respectively. The R^2 values of all carbons indicate the applicability of this equation. The calculated values q_e found from Pseudo first order equation are lesser than the experimental q_e for all carbons (Table 3). The experimental data are tested for pseudo second order equation (Ho and McKay, 1999). The plot of t/q_t against time gives a straight line (Fig. 8). Pseudo second order equation:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \tag{15}$$

k_2 is the rate constant of sorption (g mg^{-1} min^{-1}), q_e is the amount of adsorbate at equilibrium (mg g^{-1}), q_t is the amount of adsorbate on the surface of the adsorbent at any time, t (mg g^{-1}).

The R^2 values of all carbons are found close to unity which obviously indicates that the adsorption of AA on activated carbons and CAC follows Pseudo second order. A normalized standard deviation, Δq (%) is calculated to compare the efficiency of adsorption isotherms

$$\Delta q\ (\%) = 100 \times \left\{ \frac{\left(\sum \left[\frac{(q_t^{exp} - q_t^{cal})}{q_t^{exp}} \right]^2 \right)^{\frac{1}{2}}}{(n-1)} \right\} \tag{16}$$

q_t^{exp} is the experimental value of amount adsorbed at different time t and q_t^{cal} is the calculated value of amount adsorbed at different time t n is the number of observations. The Δq (%) values of pseudo second order are small and it shows that the adsorption of AA on activated carbons and CAC follows pseudo second order kinetics. The calculated values q_e found from pseudo second order equation agreed well with experimental q_e for all carbons (Table 3). This suggests that adsorption of AA on activated carbons (IASC, GSC, TSC, ASC and CSC) and CAC obeys pseudo second order kinetics more efficiently than other kinetic models.

3.7. Intra-particle diffusion model

The intra-particle diffusion is tested by applying the intra-particle diffusion model.

$$q_t = K_p t^{1/2} + C \tag{17}$$

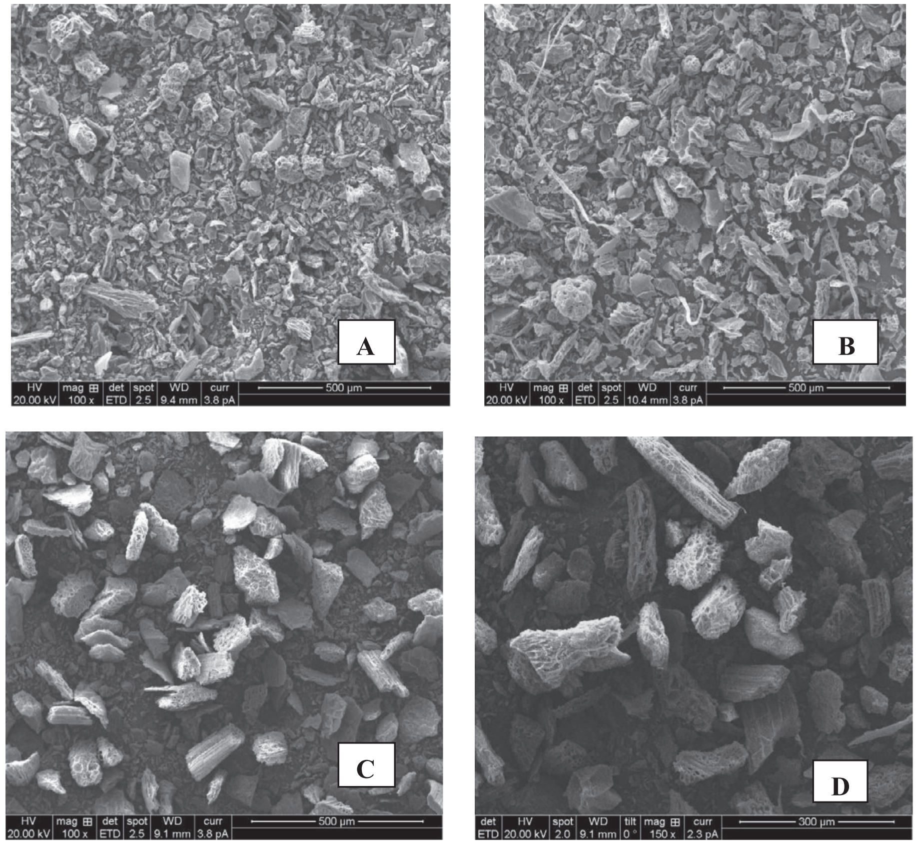

Figure 9 SEM images of IASC and GSC before (A, C) and after adsorption (B, D).

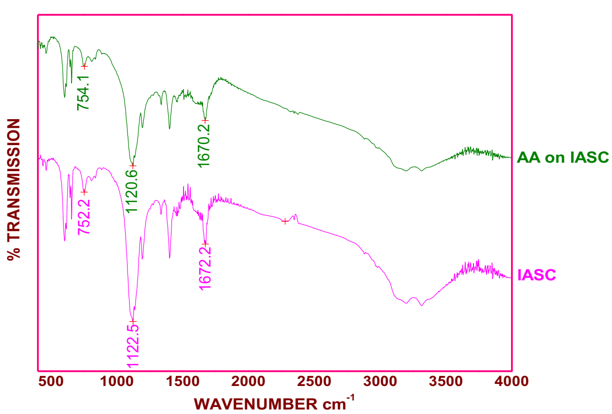

Figure 10 FT-IR spectrum of AA on IASC and IASC.

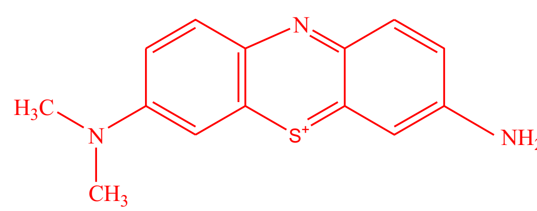

Figure 11 Structure of Azure A dye.

q_t is the amount of dye adsorbed at time t. C is the intercept and K_p is the intra-particle diffusion rate constant (mg g^{-1} -min$^{1/2}$). The correlation coefficient (r) values of all carbons indicate the existence of intra-particle diffusion process. The intercepts give an idea about boundary layer thickness. The larger the intercept, the greater is the boundary layer effect (McKay, 1983). The values of intra-particle diffusion rate constant (K_p) are calculated from the slope of the curve. The values of correlation coefficient (r), K_p and intercept are shown in Table 2.

3.8. SEM analysis

The surface morphology of the best two activated carbons IASC and GSC before and after adsorption of AA dye is shown in Fig. 9. Before adsorption of AA both the carbons appeared as fine structures (Fig. 9A and C). In contrast, after adsorption of AA, the porous structures of both the carbons disappeared (Fig. 9B and D). This indicates that AA dye molecules are strongly adsorbed by the adsorbents IASC and GSC.

3.9. FT-IR study

The Fourier Transform Infra-Red (FT-IR) spectra of AA loaded IASC and unloaded IASC are shown in Fig. 10. The peak of unloaded IASC at 752.2 may be due to N-H bending. The peak at 1122.5 may be due to C=S stretching and peak at

1672.2 may be due to C=N stretching. The FT-IR spectra of AA loaded IASC indicate that there is a slight shift in the frequency of peaks at 754.1, 1120.6, and 1670.2 which may be due to adsorption of AA on the surface of IASC. The functional groups present on the surface of IASC are responsible for adsorption of AA (Fig. 11).

4. Conclusion

The percentage removal of AA by activated carbons (IASC, GSC, ASC, TSC, CSC) and CAC is found to increase with an increase in contact time, dose, pH and decreases with an increase in initial concentration and particle size. The experimental results agreed well with Freundlich, Langmuir and Redlich–Peterson isotherms and follow pseudo second order kinetics. The adsorption capacities of all activated carbons are studied and IASC, GSC possess more adsorption capacity than ASC, TSC and CSC. The adsorptions of AA on the surfaces of adsorbents are confirmed from FT-IR analysis and SEM analysis. The adsorption capacity of AA on CAC is higher than other activated carbons. However the Activated carbons prepared from agricultural wastes could be used as an adsorbent substitute to CAC in the removal of AA from waste water.

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